भारतीय मानक

Indian Standard

IS 1448 (Part 126) : 2023

पेट्रोलियम और इसके उत्पादों की परीक्षण पद्धतियाँ

भाग 126 कच्चे और पके हुए पेट्रोलियम कोक मे राख़ की मात्रा का निर्धारण

(पहला पुनरीक्षण)

Methods of Test for Petroleum and its Products

Part 126 Determination of Ash Content in Raw and Calcined Petroleum Coke

(First Revision)

ICS 75.160.30

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January 2023 **Price Group 4**

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (excluding bitumen and hydrogen) Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Part 126) (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (excluding bitumen and hydrogen) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The ash content is one of the properties used to evaluate raw or calcined petroleum coke and it indicates the amount of inorganic impurities present. Acceptable ash content varies with the intended use. The method involves initial preparation of sample for analysis and subsequent gravimetric analysis.

This standard was originally published in 1988 was based on ASTM D 4422-84 'Standard test method for ash in analysis of petroleum coke'.

In this first revision, a clause for references has been included and test temperature has been increased from 700 °C - 750 °C to 700 °C - 900 °C. Further amount of sample has been modified to 50 g.

This standard remains technically equivalent to ASTM D 4422.

The composition of the Committee responsible for the formulation of this standard is listed in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS
PART 126 DETERMINATION OF ASH CONTENT IN RAW AND CALCINED PETROLEUM COKE

(First Revision)

1 SCOPE

This standard covers the determination of the ash content of raw and calcined petroleum coke.

2 REFERENCES

The standards listed below contain provisions which, through reference in text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves *(fourth revision)*
IS 1447 (Part 4) : 1989	Petroleum and its products methods of sampling: Part 4 Sampling of petroleum coke for laboratory analysis (*first revision*)

3 PRINCIPLE

A representative sample of raw or calcined petroleum coke is dried, ground, and ashed in a muffle furnace at 700 °C to 900 °C. The residue or ash is expressed as a percentage of the dry raw or calcined petroleum coke.

4 INTERFERENCES

4.1 Sulphur content present in petroleum coke reacts with alkaline residue and give abnormal results. Therefore, furnace shall have a provision for air flow, so as to oxidize sulphur content properly.

4.2 Care shall be taken while sample preparation to avoid error in test results. Improper dividing, sieving, and crushing equipment and some muffle furnace lining material may contaminate the sample.

5 APPARATUS

5.1 Crucibles — Glazed porcelain or platinum, 20 ml to 30 ml capacity. Only platinum crucible shall be used, if ash needs to be analysed further.

5.2 Muffle Furnace — With temperature control between 700 °C and 900 °C and equipped with means to regulate air flow.

5.3 Analytical Balance — Capable of weighing with accuracy up to 0.1 mg.

5.4 Drying Oven — Capable of controlling and maintaining temperature in the range of 105 °C to 115 °C.

5.5 Desiccator — Suitable to cool the crucible.

6 PREPARATION OF APPARATUS

Required air flow rate of the muffle furnace shall be ensured, while installation or at the time of major maintenance. Flow rate is considered suitable, if a sample is retested at higher flow rate and the test results are found to be within the repeatability limits.

7 SAMPLE PREPARATION

Take a test sample as per the procedure described in IS 1447 (Part 4). Crush the test sample to pass 6.3 mm sieve and further reduce sample to pass 0.85 mm sieve [(*see* IS 460 (Part 1)]. Finally prepare (50 ± 1) g of sample with 250 micron sieve [*see* IS 460 (Part 1)], set the oven temperature to 110 °C, and dry the sample till a constant mass is obtained. If the initial raw or calcined petcoke sample is in large quantity, it is to be reduced to the amount required and air dried, if it is in wet condition to avoid caking.

8 PROCEDURE

8.1 Take (1.0 ± 0.1) g of raw or calcined petcoke sample that is passed through 250 micron sieve, in a pre-dried and coded crucible. Record the weight of coded crucible and crucible with sample up to 0.000 1 g.

8.2 Place the crucible in the muffle furnace at room temperature. Set the furnace temperature between 700 °C to 900 °C and start the furnace heating. Record the weight until constant mass (± 0.2 mg) is obtained.

8.3 Allow the furnace to cool to 125 °C to 150 °C and transfer the crucible to the desiccator for further cooling.

8.4 Weigh the crucible to the nearest 0.000 1 g and calculate the ash mass percent.

9. CALCULATION

Calculate the ash mass percent in the analysis sample as follows:

$$\text{Ash, mass percent} = \frac{A-B}{C} \times 100$$

where

A = mass of crucible and residue, in g;
B = mass of empty crucible, in g; and
C = mass of the sample for analysis, in g.

10 REPORT

10.1 Report the ash content as mass percent to the third decimal, when tested in duplicate and report average result.

10.2 Report the ash content as mass percent to the second decimal, when only tested for a single time.

11 PRECISION

Precision of the ash test, based on petroleum coke ash test results of inter laboratory correlation study (three laboratories), are as follows:

11.1 Repeatability

Duplicate test results of same sample performed in single laboratory by following standard test procedure using same machine and by different operator shall not be vary more than 0.03 mass percent.

11.2 Reproducibility

Two test results of same sample performed in different laboratory by following standard test procedure using different machine and by same operator shall not be vary more than 0.06 mass percent.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (excluding bitumen and hydrogen) Sectional Committee, PCD 01

Organization	*Representative(s)*
Indian Institute of Petroleum, Dehradun	Dr Anjan Ray (***Chairperson***)
Air Headquarters, Ministry of Defence, New Delhi	WG CMDR S. A. Tawab WG CDR D. Anandraj Roy (*Alternate*)
All India Instrument Manufacturers and Dealers Association, Mumbai	Prof G. M. Rao
Bharat Oman Refineries Limited, Bina	Shri Anand Pratap Raghav Shri B. Sridhar (*Alternate*)
Bharat Petroleum Corporation Limited, Mumbai	Shri R Subramanian Shri C. Shanmuganathan (*Alternate*)
Castrol India Limited, Mumbai	Shri Raman Rai
CSIR - Central Institute for Mining and Fuel Research, Dhanbad	Shri P. K. Singh Shri S. R. K. Rao (*Alternate* I) Shri S. Dutta (*Alternate* II)
Central Institute of Plastics Engineering and Technology, Bhuwaneshwar	Dr Smita Mohanty
Central Revenue Control Laboratory, New Delhi	Dr T. A. Sreenivasa Rao
Chennai Petroleum Corporation Limited, Chennai	Shri H. Ramakrishnan Dr V. Selvavathi (*Alternate*)
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Directorate General of Quality Assurance, Ministry of Defence, Kanpur	Shri Sachin Vinayak Zope
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Indian Oil Corporation (R and D Centre), Faridabad	Shri V. Kagdiyal Dr S. K. Majumdar (*Alternate*)
Indian Oil Corporation Limited - Refineries and Pipelines Division, New Delhi	Dr Ashutosh Mishra Dr Shashi Pal Singh (*Alternate*)
Indraprastha Gas Limited, New Delhi	Shri P. K. Pandey Shri Rahul Nigam (*Alternate*)
Lubrizol India Limited, Mumbai	Shri Anil Mane Shrimati Reena Kuril (*Alternate*)
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Ministry of Railways, Lucknow	Shri Rajesh Srivastava Shri Sonam Gupta (*Alternate*)
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Numaligarh Refinery Limited, Golaghat	Shri Sobhan Konwar Shri Anjan Kalita (*Alternate*)
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Oil and Natural Gas Corporation Limited, New Delhi	Shri Gour Mohan Dass Shrimati. Leena John (*Alternate* I) Shri Dinesh S. R. Reddy Kakuturi (*Alternate* II)
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Petroleum and Natural Gas Regulatory Board, New Delhi	Shri Noor Khan Shri Anil Meghani (*Alternate*)
Reliance Industries Limited, Mumbai	Shri Pramod Mall
Shriram Institute for Industrial Research, Delhi	Dr Mukesh Garg Dr Rajesh Kumar (*Alternate*)
Director General, BIS, New Delhi	Shrimati Meenal Passi, Scientist 'F'/Senior Director and Head (Petroleum, Coal and Related Products) [Representing Director General (*Ex-officio*)]

Member Secretary
Shri Aridaman
Scientist 'B'/Assistant Director
(Petroleum, Coal and Related Products), BIS

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